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from Soy-Based Dimer Acids and Different Amides

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Synthesis and Characterization of Polyamide Resins from Soy-based Dimer Acids and Different Amides

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Synopsis

A series of soy-based polyamides with different dimer acids and diamines were synthesized using a condensation polymerization technique. The molecular weight of polyamides prepared from 1,4-phenylenediamine increase greatly with a reaction temperature above 260°C. The physical properties of the polyamides, such as glass transition temperature (T_g), melting point (T_m), decomposition temperature (T_d), crystalline behavior, and mechanical strength strongly depend on their molecular weight and flexibility of diamines used. The aromatic-based polyamides have a higher T_g , T_m , T_d and stronger mechanical strength than that of aliphatic-based polyamides. X-ray diffraction patterns of the samples indicate that all the resins synthesized present a typical semicrystalline morphology. Polyamides made from hydrogenated dimer acid possess lower T_g and higher mechanical strength compared with polyamides from unsaturated dimer acid with different dimer and trimer ratios. These results are analyzed and

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discussed in accordance with the influence of rigid aromatic segments and the microstructure of different dimer acids.

Key Words: Polyamide, dimer acid, aromatic, soy-based, condensation.

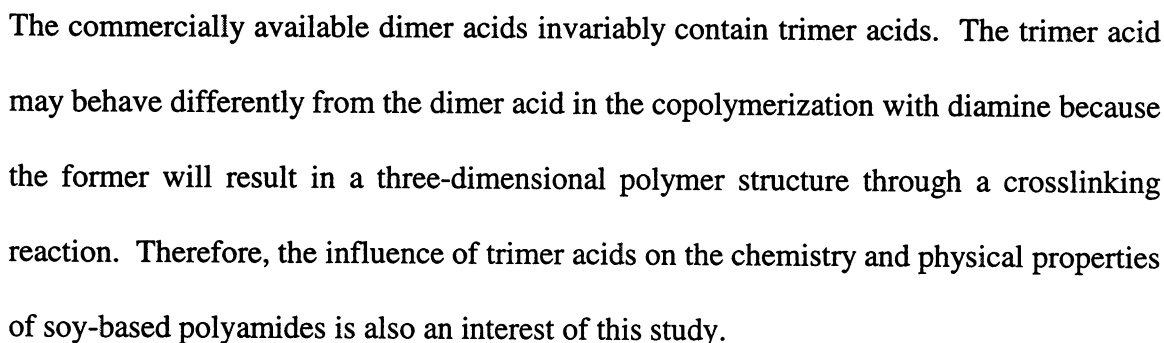
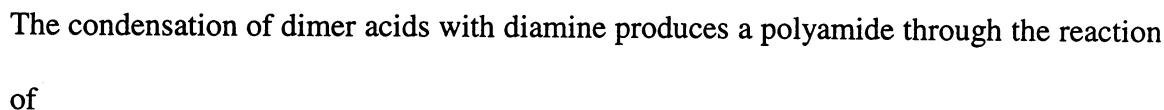
Introduction

Soy-based dimer acids have been traditionally used to synthesize and formulate hot-melt adhesives, flexographic inks, functional coatings, and engineering materials [1-10]. Because of their wide applications, the investigations of soy-based polyamides are growing. There are many advantages of using soy-based dimer acids as starting materials compared with using other monomers because they are nontoxic, less expensive, and potentially biodegradable. The polyamides prepared by condensation polymerization of soy-based dimer acids with different diamines show many interesting physical properties, such as forming extreme strong bonds to most solid surfaces, having a sharp melting temperature at 90-120°, reacting fast with epoxy resins, etc. Although many novel formulations and synthesis techniques related to dimer acid-based polycondensation products were disclosed in a recent series of patents [11-13], systematic studies focusing on the crystalline structure, and thermal and mechanical behaviors of these resins are relatively few. On the other hand, despite extensive research of formulations using different aliphatic amines, the condensation polymerization of a dimer acid with aromatic amines has not been extensively studied. It is well-known that incorporating aromatic segments into a flexible matrix can add exceptional mechanical strength and thermal

stability, compared with pure aliphatic segments [14-16]. The results are attributed to the rigid segmental structure and related crystal morphology. Thus, the study of dimer acid-based polyamides with aromatic segments not only possesses value for scientific research, but also can expand commercial utilization of this polymer.

The application of soy-based polyamide as a toner resin for office copiers and laser printers has been developed in our laboratory recently [17]. A potential advantage to using this particular resin in copy toner formulas is that the soy-based polyamide may be biodegradable so that it can be easily deinked by adding certain enzymes during the deinking process. Toners have been typically designed to have a T_g (glass transition temperature) ranging from 50 to 65°C [18]. If the T_g of the resins is below 50°C, the particles tend to stick together, making the material unacceptable as a toner. However, commercially available soy-based polyamides usually have a glass transition temperature much lower than 50°C. To synthesize a soy-based toner with a required T_g , a dimer acid with unsaturated segments, or an aromatic diamine may be used. The influence of monomer structure and flexibility on the glass transition temperature of soy-based polyamides was examined in this study.

The soy-based dimer acids can be prepared by condensation of two unsaturated fatty acids such as oleic acid (10-59% in soybean oil) or linoleic acid (28-66% in soybean oil) in the presence of a clay or other catalyst [19],



Experimental

Materials: Dimer acid was supplied by the Emery Group of Henkel Corporation. Empol 1007 and 1018 dimer acids are both yellowish viscous liquids at room temperature with dimer and trimer ratios at 97:2 and 75:19, respectively. Hydrogenated dimer acid

and all of the diamines were purchased from Aldrich and used as received. Their acronyms, reported purity, molecular structure, and melting point are listed in **Table 1**.

Polymer synthesis: The condensation polymerization was used for the polymer synthesis. The polymerization conditions and procedures were the same except that different temperatures and monomers were used. A typical example of polymerization (for sample D1007P) is given as follows: 100 g of dimer acid (equivalent to 0.353 mol acid groups) was charged into a 250-ml three-necked round bottom flask equipped with a mechanical stirrer, a thermometer, and a nitrogen inlet. The monomer was first heated to 80°C under a nitrogen atmosphere and vigorous stirring; then 19.2 g of 1,4-phenylenediamine (equivalent to the total acid groups) were added. The reactants were gradually heated to 260°C in 3 hours and kept at this temperature for another 5 hours under vigorous stirring. Then the system was allowed to cool down to room temperature. The next day the reactants were heated again to 260°C and subjected to vacuum of approximately 2 mm Hg for 4 hours. The product was then discharged from the flask.

Determination of molecular weight: The number average molecular weight of the polyamides synthesized was determined from the combined content of acid and amine end groups [20]. Carboxylic end groups were determined by titration of a 0.5% polyamide solution (pyridine as the solvent) with 0.10 N methanol sodium hydroxide. Phenolphthalein was used as the indicator. Amine end groups were determined by titration with freshly standardized 0.10 N propanol hydrochloric acid. Brom cresol green solution was used as the indicator. For all the titration experiments, at least three

replicates including a blank titration of solvent were collected for determining the reproducibility.

The number average molecular weight, M_n , was calculated based on the following equation [20]:

$$M_n = \frac{W}{(C_A V_A + C_B V_B)} \left(\frac{N_m}{1} + \frac{N_d}{2} + \frac{N_t}{3} \right)^{-1}$$

where W is the weight of solid sample titrated; C_A , C_B and V_A , V_B are the concentrations and actual volumes used from standard solutions of sodium hydroxide and hydrochloric acid, respectively; and N_m , N_d , and N_t are the percent concentrations of the monomers with one, two, and three functional groups (acid and amide) in the products.

The repeat unit structure of polymers and their number average molecular weights are listed in **Table 2**.

Wide-angle x-ray diffraction (WAXD) measurement: WAXD patterns of polyamide resins were obtained with a Diano-XRD 8000 x-ray diffractometer using nickel-filtered Cu-K α radiation. The tube current and voltage are 20 mA and 40 KV, respectively. Measurements were scanned between 0 and 5° 2 θ angle at 0.04° steps. The counting time was 1 second per step.

Differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA): DSC measurements were performed on a SEICO DSC equipped with a thermal analysis station. TGA measurements were carried out on a SEICO TGA. Both DSC and TGA measurements were performed under nitrogen at a 20°C/min heating rate.

Stress-strain analysis: Uniaxial stress-strain measurements of polyamide films were conducted (ASTM D 882-91) using an Instron Model 1122 at 20°C and 50% relative humidity. All films were 15 mm in width and 200 (± 50) μm in thickness. A 50-mm gauge length and a 5 mm/min crosshead speed were employed.

Results and Discussion

Influence of reaction temperature on polyamides' molecular weight and physical properties: Synthesis of polyamides using dimer acid and diamine is a typical condensation polymerization, which usually takes place through the elimination of water molecules at certain reaction conditions. The molecular weight of dimer acid-based polyamides can be affected by reaction time, temperature, monomer ratio, catalyst, etc. If other parameters remain a constant, the reaction temperature, which is directly related to the rate of distillation of condensing water, will significantly affect both the polymerization rate and the molecular weight. To further explore this factor, reaction temperatures of dimer acid 1007 with 1,4-phenylenediamine versus their number average molecular weights are plotted in **Figure 1**. It is evident that the molecular weight of polyamide with the aromatic diamine increases with increasing reaction temperature. Above 260°C, there is a rapid increase in molecular weight with increasing temperature. This may indicate that the polycondensation reaction resulting from eliminating water in this system can proceed much faster above 260°C. The result is consistent with several recent reports that the optimum reaction temperature of dimer acid and aliphatic diamines is in the range of 260 to 300°C [11-13]. It may also imply that polycondensations of both aliphatic and aromatic diamines with dimer acids have a similar reaction mechanism.

The wide-angle x-ray diffraction diagrams of polyamides with aromatic diamine synthesized at different temperatures are shown in **Figure 2**. All samples have a relatively sharp diffraction peak at $2\theta = 20^\circ$ and a broad shoulder peak around $2\theta = 40^\circ$. There is no significant variation observed with reaction temperature, except the peak width of the sample prepared at 160°C is a little broad. Compared with the X-ray diffraction diagrams of Nylon's homopolymers, polyblends and block copolymers [21], it was found that dimer acid-based polyamides possess similar x-ray diffraction patterns with Nylons, and can be identified as the semicrystalline polymers. The thermal analysis data can further confirm this conclusion.

Figure 3 presents the thermal behavior of polyamides versus their molecular weight. It is seen that the glass transition temperature (T_g), melting temperature (T_m), and enthalpy of fusion (ΔH) gradually increase with the increase in molecular weight. The T_g and T_m of the sample with the largest molecular weight are about 20°C higher than that of the sample with the lowest molecular weight. As T_g reflects the Brownian motion of amorphous chains, and T_m represents the chain motion in crystals, higher T_g and T_m may indicate stronger restrained amorphous segments and higher ordered crystals due to an increased molecular weight in the samples. The increase in the enthalpy of fusion with an increase in molecular weight supports the idea that higher molecular weight polyamides may have a higher crystallinity as reported previously [21]. The results clearly demonstrate that the polycondensation reaction temperature can greatly influence the molecular weight of polyamide, and in turn, affect its T_g , melting temperature, and crystalline behavior.

The variation of mechanical properties of polyamides with their molecular weight is plotted in **Figure 4**. Evidently, Young's modulus (E), stress at break (σ), and strain at break (ϵ) all increase with increasing molecular weight. It is well-known that the mechanical properties of solid polymers, especially for semicrystalline polymers, rely closely on their amorphous and crystal morphology [23]. A higher mechanical performance of the sample may indicate that there is a better organized crystal and a less defective amorphous structure through their longer molecular chains. On the other hand, the samples with lower molecular weight could cause lower crystallinity and have more end groups concentrated into their amorphous phase, resulting in poor mechanical performance.

The influence of aromatic and aliphatic diamines on polyamides' physical properties: It has been established that polyamides with aromatic segments could possess better thermal and mechanical performances than that of aliphatic segments [24, 25]. For further comparison of the influence of both aromatic and aliphatic diamines on the physical properties of dimer acid-based polyamides, ethylenediamine, hexamethylenediamine, and 1,4-phenylenediamine were selected as the monomers for preparation of polyamide resins. The polymers' segmental structure and their molecular weight are listed in **Table 2**. It can be seen from **Table 2** that the molecular weight of D1007E, D1007H, and D1007P (prepared at 260°C) are basically in the range of 10^4 g mol⁻¹.

The DSC curves of the three samples are presented in **Figure 5**, and related thermal properties are given in **Table 3**. First, it can be seen that the T_g of D1007H

(hexamethylenediamine-based polyamide) is only 7.0°C; by contrast, the T_g of D1007P (1,4-phenylenediamine-based polyamide) is 62.8°C. As the T_g represents the chain segments' motion in amorphous phase, the higher T_g of D1007P suggests that the segments' motion in the aromatic diamine could be strongly restrained by their rigid molecular structure. On the other hand, as hexamethylenediamine is more flexible than ethylenediamine, a higher T_g value for D1007E indicates that the flexibility of segments in the aliphatic diamine monomer still plays an important role in effecting their T_g .

Similarly, with the variety of glass transition temperature, the melting temperature of three samples increases with a decrease in diamine flexibility also. Especially, the melting temperature of D1007P (131.8°C) is almost 50°C higher than that of D1007H (81.6°C). The higher melting temperature in D1007P may indicate that its aromatic segments in the crystal phase are strongly interacting and need more energy to change their configuration and initiate them to move. The lower melting temperature of D1007H may result from its flexible chain structure. Besides, D1007P's thermal decomposition temperature from TGA data is about 20°C higher than that of D1007E and D1007H, respectively, indicating its excellent heat-resistant properties.

Interestingly, it was found from **Table 3** that the enthalpy of fusion of the three samples decreases with the increase in flexibility of diamine. The more flexible the segment, the higher the value of enthalpy of fusion. According to calorimetry theory [26], for a semicrystalline polymer, the degree of crystallinity can be calculated by the ratio of the enthalpy of fusion of the sample to that of its perfect crystalline polymer. Because aromatic-based polyamide has stronger segments interaction and lower

flexibility than that of an aliphatic one, it is expected that hexamethylenediamine-based polyamide should move easily at melting point, which results in a lower enthalpy of fusion in its perfect polymer crystal. A higher enthalpy of fusion of an aliphatic-based polyamide sample from the experiment (see **Table 3**) and an assumed lower enthalpy of fusion of its perfect crystals suggest that the hexamethylenediamine-based polyamide may have the highest crystallinity. This conclusion is also supported by x-ray diffraction data (**Figure 6**), i.e., sample D1007H has a relative sharper diffraction peak ($2\theta = 20^\circ$) compared with D1007E and D1007P.

The stress-strain curves of the three samples are shown in **Figure 7**. As expected, D1007P has the highest Young's modulus and stress at break; D1007E is intermediate; and D1007H has the lowest modulus and stress at break (see **Table 3**). Furthermore, D1007P and D1007E appear to be brittle in character, where there is no obvious yield point and ductile region observed. In contrast to D1007P and D1007E, D1007H possesses a typical ductile fracture feature, where there exists a clear yield point and a longer deformation region. Its average strain at break reaches to 7.1%. The results may still be attributed to the rigid aromatic and flexible aliphatic chain segments in their amorphous phases, respectively. It is well-known that tensile fracture in semicrystalline polymers generally occurs in their amorphous region, because heterogeneities such as chain ends and ruptured bonds most likely concentrate in the amorphous region, and these could lead to "disruption points" and stress concentrations [27]. The higher mechanical strength and Young's modulus in D1007P are evidently related to its rigid aromatic segments, which can effectively resist formation of stress-induced microcracks or crazes

during the deformation process. As for D1007H, plastic deformation occurs as soon as the stress is applied to it, due to its flexible segments, and leads to a decrease in mechanical properties.

The effect of different dimer acids on polyamides' physical properties: In order to study the effect of the molecular structure of a dimer acid monomer on the polymer's molecular weight and physical behavior, dimer acid 1018 (79% dimer, 15% trimer, and 6% monomer acid) and hydrogenated dimer acid (98%+ dimer acid) were also reacted with 1,4-phenylenediamine for preparing polyamides. Their segmental structure and molecular weight are compared in **Table 2**. At the same polymerization conditions, it can be seen that D1007P and D1018P have a similar molecular weight and HYDAP's molecular weight is higher than that of D1007P and D1018P. Theoretically, compared with D1007P, D1018P is expected to have more crosslinks, branched structures, and relatively larger molecular weight polydispersity, due to its higher content of trimer and monomer acids. Because both the contents of trimer and monomer acids are higher in 1018 than in 1007 where the trimer acid may result in an increase and monomer acid may cause a decrease in molecular weight, it was not surprising that a similar molecular weight was obtained for D1007P and D1018P. It should be mentioned that the end group analysis method used to calculate molecular weight in this case is only based on the amount of end groups in the system; it cannot represent the molecular structure factor, let alone the polydispersity. The higher molecular weight of HYDAP may be attributed to its higher purity of dimer acid, which could construct the fine and long liner macromolecular chain.

Table 4 lists the thermal and mechanical properties of HYDAP, D1018P, and D1007P. First, we can see that HYDAP's T_g is almost 20°C lower than that of D1018P and D1007P. This result may be attributed to the fact that the hydrogenated dimer acid has less rigid saturated segments [28], because an unsaturated dimer acid may likely form a conjugated molecular complex [29] through its double bonds and restrict the segments' motion accordingly. As for D1018P and D1007P, it is reasonably assumed that, within experiment error, they have the same T_g . Although the hydrogenated dimer acid gives a significantly lower T_g than the nonhydrogenated one, the T_m for these two types of polyamides is almost the same. However, we did find that the melting temperature of D1018P was 6°C lower than that of D1007P and HYDAP. The lower melting temperature may imply a lower ordered crystalline morphology due to its more heterogeneous molecular structure (more trimer acid content) in D1018P. The lower enthalpy of fusion of D1018P may further confirm this point. For more details about their crystalline structure, we conducted x-ray diffraction measurements for these samples; unfortunately, we did not find more useful information to discern their difference except a common sharp diffraction peak at $2\theta = 20^\circ$.

It can also be seen from **Table 4** that HYDAP possesses the higher Young's modulus and stress at break compared with D1007P and D1018P. This may be a result of its large molecular weight and relative homogeneous crystalline structure. Comparing D1018P with D1007P, the higher tensile stress and Young's modulus for D1018P may be attributed to the more microcrosslinking structure in its amorphous phase.

Conclusions

The molecular weight of soy-based polyamides through polycondensation reaction of different dimer acids and diamines can be effectively controlled by controlling the reaction temperature. The optimum reaction temperature that can synthesize higher molecular weight polymers is in the range of 260 to 300°C. Polyamides prepared from 1,4-phenylenediamine possess higher T_g , T_m , and T_d and show stronger mechanical strength than that of aliphatic polyamides. The results can be attributed to the influence of flexibility of aromatic and aliphatic diamine segments on the polymers' crystallization behavior and different morphology. X-ray diffraction data indicate that dimer acid-based polyamides are typical semicrystalline polymers. A polyamide made from a hydrogenated dimer acid has a lower T_g and higher mechanical strength compared with polyamides made from an unsaturated dimer acid. This indicates that the flexibility of the dimer acid and its macrostructure, such as the content of monomer and trimer acids, still play a role in effecting the polymer's properties.

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Figure Caption:

Figure 1. Molecular weight of D1007P versus polycondensation reaction temperature.

Figure 2. X-ray diffraction patterns for D1007P synthesized at different temperatures.

Figure 3. Molecular weight versus glass transition temperature, melting temperature, and enthalpy of fusion for D1007P.

Figure 4. Molecular weight versus Young's modulus, stress at break, and strain at break for D1007P.

Figure 5. DSC diagrams for D1007E, D1007H, and D1007P.

Figure 6. X-ray diffraction patterns for D1007E, D1007H, and D1007P.

Figure 7. Stress-strain curves for D1007E, D1007H, and D1007P.

Table 1. Characteristics of the Monomers.

| Monomer | Acronym | Structure | T _m /°C |
|--------------------------------------|---------|---|----------------------------|
| Dimer Acid* | D1007 | $\text{HO}-\overset{\text{O}}{\parallel}{\text{C}}-(\text{CH}_2)_7-\text{CH}=\text{CH}-\text{C}_6\text{H}_8-\text{CH}_2-\text{CH}_2-(\text{CH}_2)_7-\overset{\text{O}}{\parallel}{\text{C}}-\text{OH}$ <p style="text-align: center;">CH₃(CH₂)₅ CH₃(CH₂)₅</p> | Liquid at room temperature |
| Dimer Acid [#] | D1018 | Same above | Same above |
| Dimer Acid Hydrogenated (98%+ dimer) | HYDA | $\text{HO}-\overset{\text{O}}{\parallel}{\text{C}}-(\text{CH}_2)_7-\text{CH}_2-\text{CH}_2-\text{C}_6\text{H}_{10}-\text{CH}_2-\text{CH}_2-(\text{CH}_2)_7-\overset{\text{O}}{\parallel}{\text{C}}-\text{OH}$ <p style="text-align: center;">CH₃-(CH₂)₅ CH₃-(CH₂)₅</p> | Same above |
| 1,4-Phenylene-diamine (99%) | P | $\text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{NH}_2$ | 143-145 |
| Hexamethylene-diamine (98%) | H | $\text{H}_2\text{N}-(\text{CH}_2)_6-\text{NH}_2$ | 42-45 |
| Ethylene-diamine (99%) | E | $\text{H}_2\text{N}-\text{CH}_2-\text{CH}_2-\text{NH}_2$ | 8.5 |

*D1007: 97% dimer, 2% trimer, and 1% monomer acid

[#] D1018: 79% dimer, 15% trimer, and 6% monomer acid

Table 2. Molecular Weight of Polymers and Their Structure.

| Polyamide | Acronym | Structure | M _n (g/mol) |
|---|-------------------|---------------|------------------------|
| Based on dimer acid 1007 and ethylenediamine | D1007E | | 10500 |
| Based on dimer acid 1007 and hexamethylenediamine | D1007H | | 10400 |
| Based on dimer acid hydrogenated and 1,4-phenylenediamine | HYDAP | | 11500 |
| Based on dimer acid 1007 and 1,4-phenylenediamine | D1007P (160°C) | | 3300 |
| | (220°C) | same as above | 7400 |
| | (260°C) | same as above | 9000 |
| | (310°C) | same as above | 26000 |
| Based on dimer acid 1018 and 1,4-phenylenediamine | D1018P | same as above | 9500 |

Table 3. Thermal and Mechanical Properties of Polyamides with Different Diamines.

| Sample | Glass Transition Temperature (°C) | Melting Temperature (°C) | Enthalpy of Fusion (J/g) | Decomposition Temperature (°C) | Young's Modulus (MPa) | Stress at Break (MPa) | Strain at Break (%) |
|---------------|--|---|---|---|--------------------------------------|--------------------------------------|------------------------------------|
| D1007E | 60.9 | 103.0 | 26.1 | 385.0 | 2017 | 27.3 | 4.4 |
| D1007H | 7.0 | 81.6 | 28.0 | 390.0 | 1322 | 14.5 | 7.1 |
| D1007P | 62.8 | 131.8 | 19.6 | 406.5 | 2116 | 33.3 | 5.3 |

Table 4. Thermal and Mechanical Properties of Polyamides with Different Dimer Acids.

| Sample | Glass Transition Temperature (°C) | Melting Temperature (°C) | Enthalpy of Fusion (J/g) | Decomposition Temperature (°C) | Young's Modulus (MPa) | Stress at Break (MPa) | Strain at Break (%) |
|--------|-----------------------------------|--------------------------|--------------------------|--------------------------------|-----------------------|-----------------------|---------------------|
| HYDAP | 44.2 | 132.1 | 21.2 | 400 | 3191 | 40.7 | 2.6 |
| D1018P | 58.4 | 125.5 | 18.1 | 406 | 2541 | 38.3 | 4.4 |
| D1007P | 62.8 | 131.8 | 19.6 | 406 | 2116 | 33.3 | 5.3 |

Figure 1.

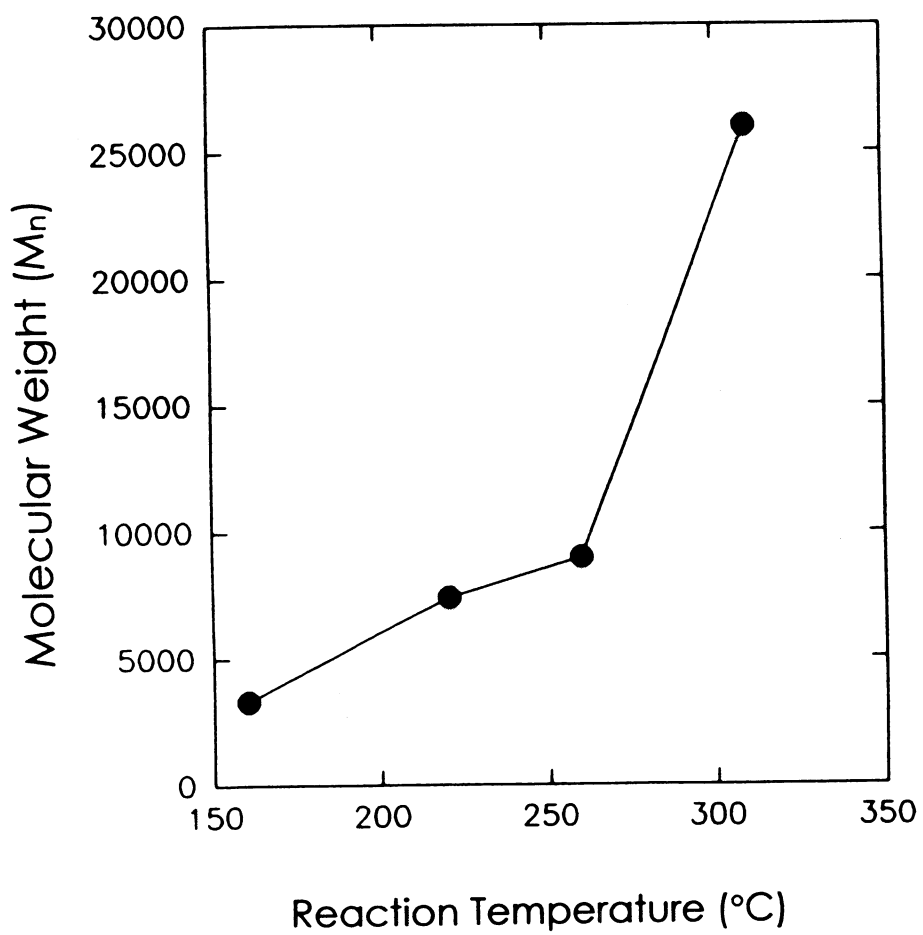


Figure 2.

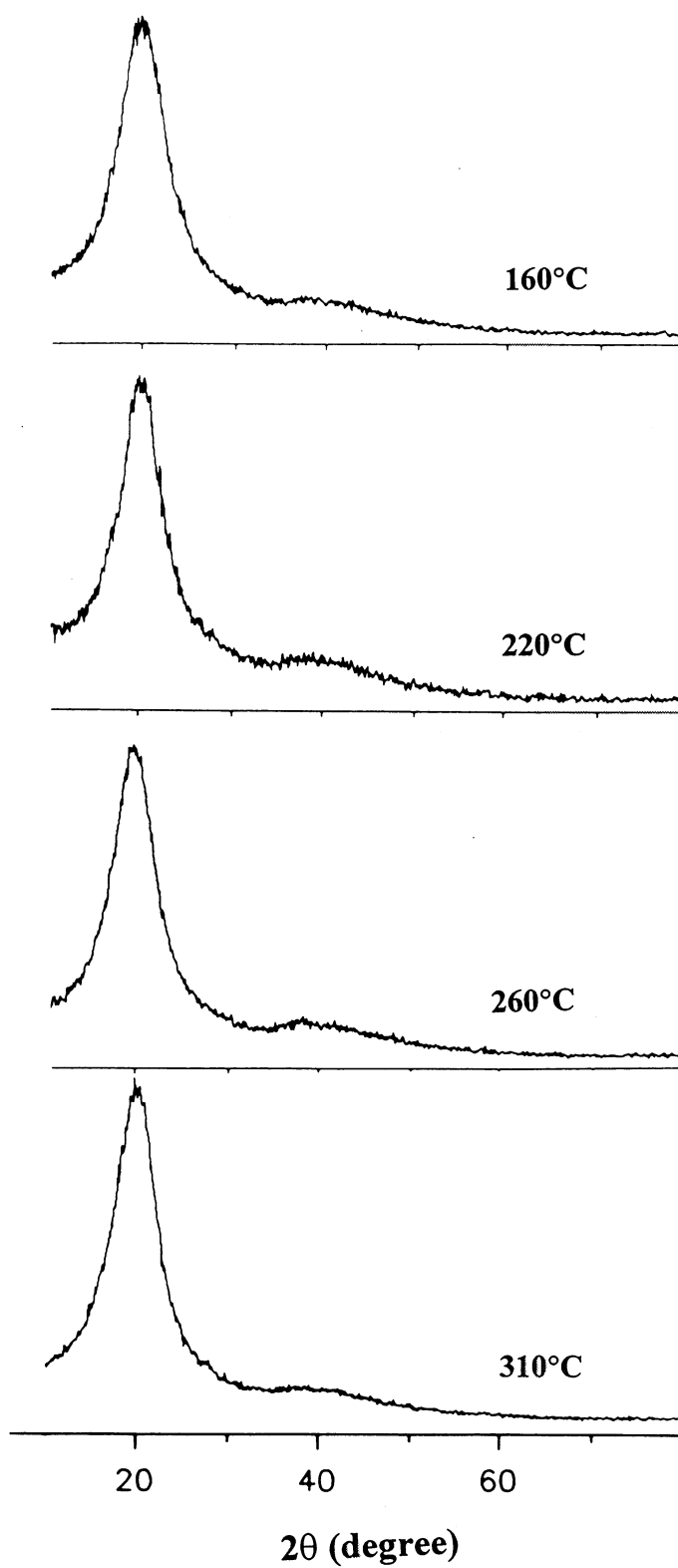


Figure 3.

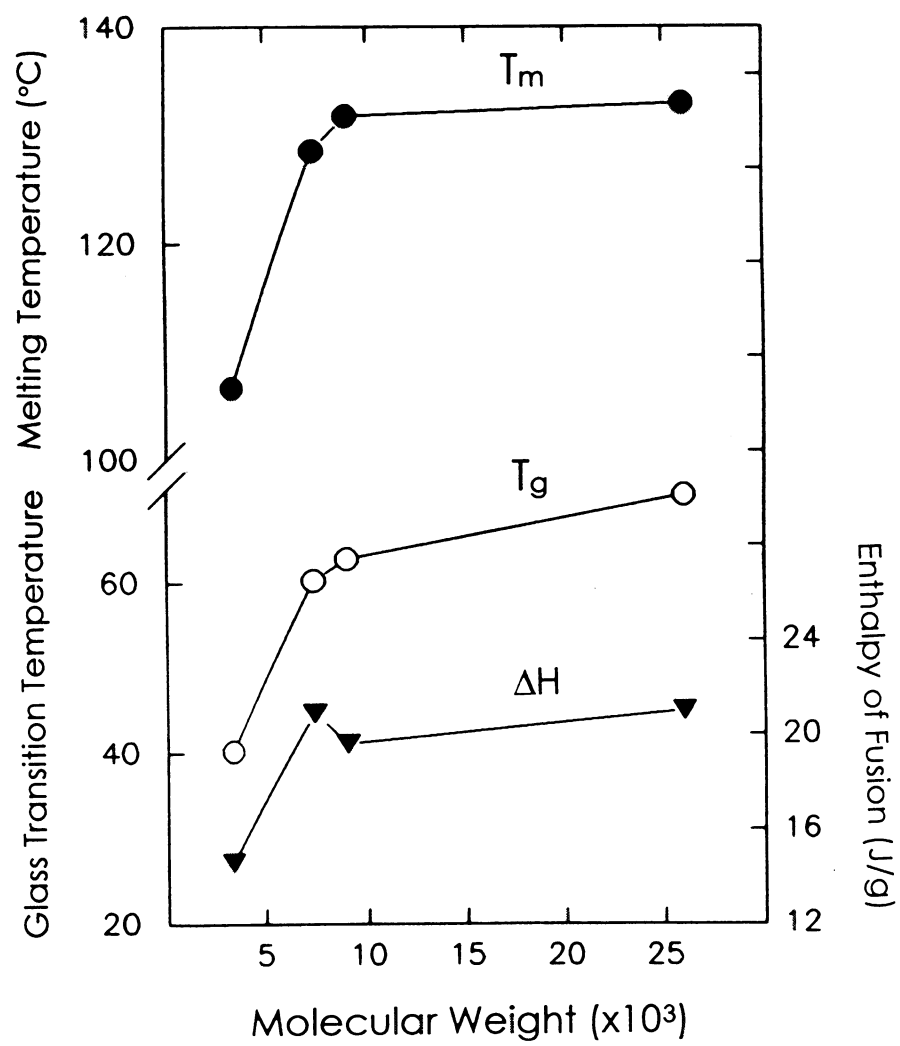


Figure 4.

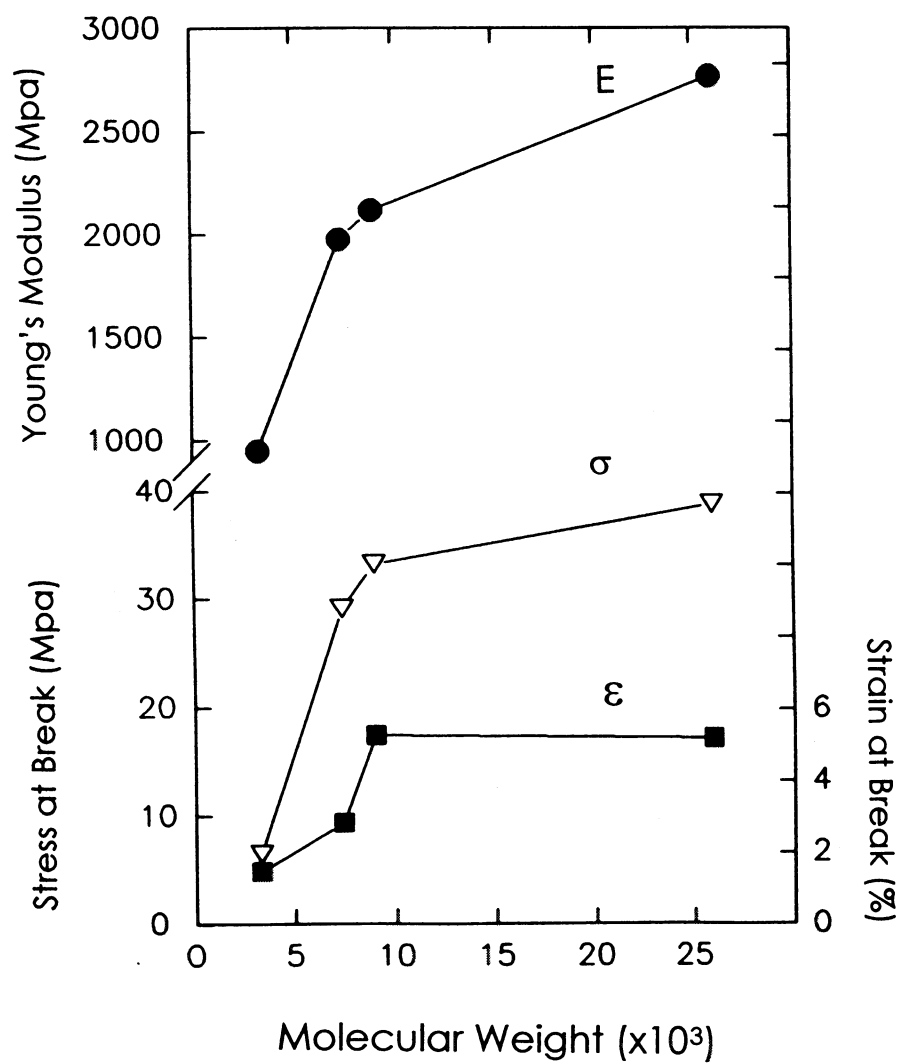


Figure 5.

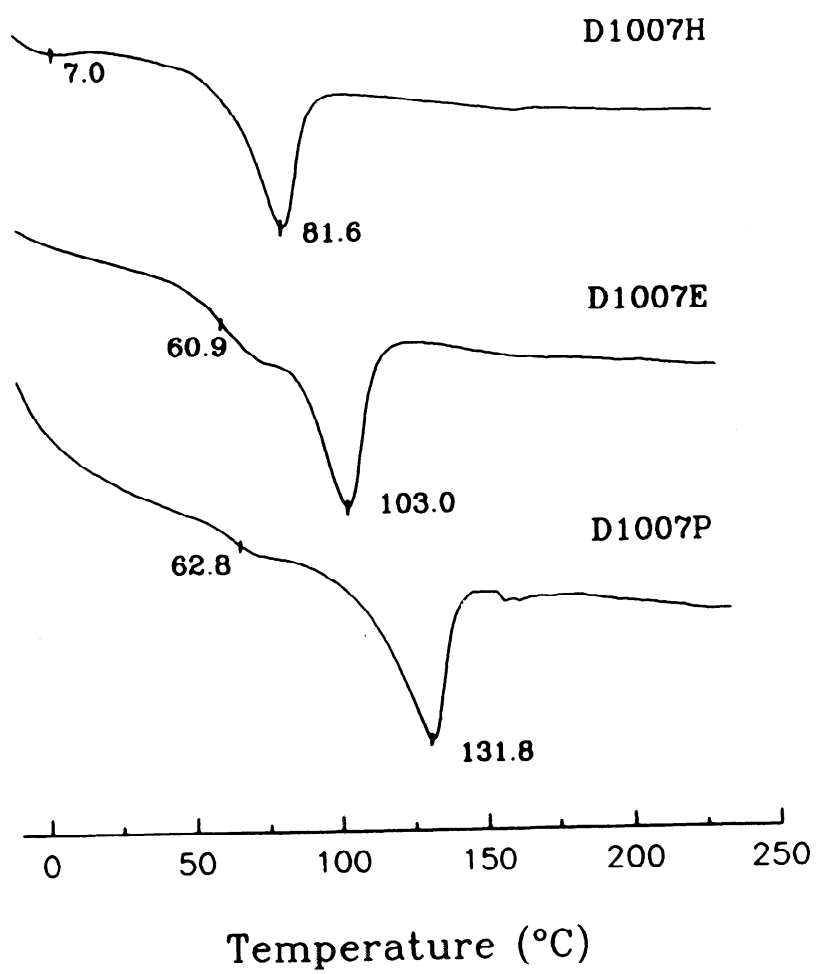


Figure 6.

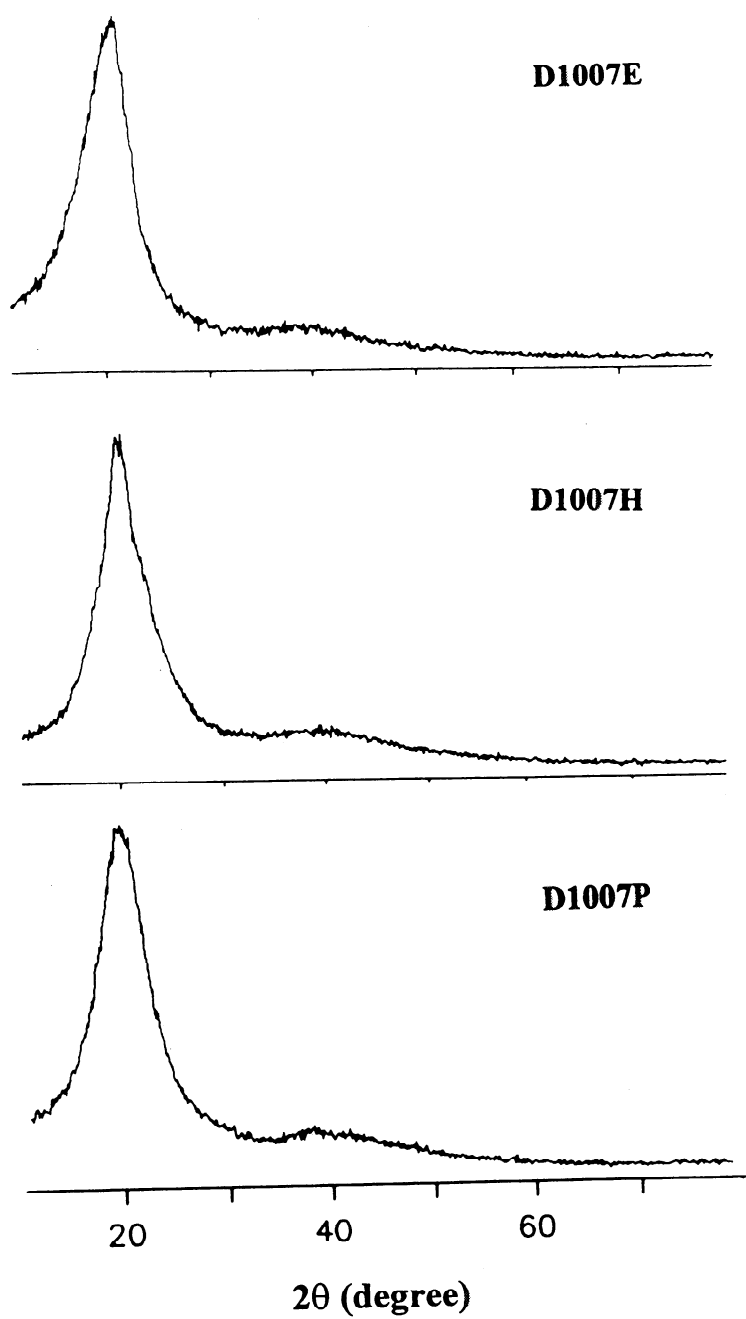


Figure 7.

